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मानक

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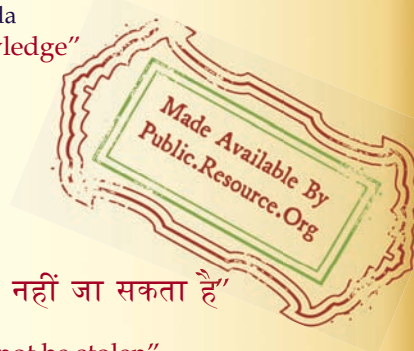
IS 11744 (1986): Phosphorus pentachloride, technical [CHD
1: Inorganic Chemicals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

SPECIFICATION FOR
PHOSPHOROUS PENTACHLORIDE, TECHNICAL

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*Indian Standard*SPECIFICATION FOR
PHOSPHOROUS PENTACHLORIDE, TECHNICAL

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Indian Standard

SPECIFICATION FOR PHOSPHOROUS PENTACHLORIDE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 5 July 1986, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Phosphorous pentachloride is mainly used as a protective chlorinating agent in the manufacture of pharmaceutical and dyestuffs.

0.3 Phosphorous pentachloride also serves as a catalyst for condensation and cyclization reactions and for improving grain structure of light metal castings and as a reagent in analytical laboratory. It is also used as dehydrating agent.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for phosphorous pentachloride, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of pale yellow fine granular powder, having an irritating odour, soluble in carbon disulphide and carbon tetrachloride.

2.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clause of Appendix A is given in col 4 of the table.

*Rules for rounding off numerical values (revised).

TABLE 1 REQUIREMENTS FOR PHOSPHOROUS PENTACHLORIDE,
TECHNICAL

(Clause 2.2)

| Sl No. | CHARACTERISTIC | REQUIREMENT | METHOD OF TEST, REF TO CL No. OF APPENDIX A |
|--------|---|-------------|---|
| (1) | (2) | (3) | (4) |
| i) | Phosphorous pentachloride (as PCl_5), percent by mass, <i>Min</i> | 97.0 | A-2 |
| ii) | Bulk density, g/ml | 0.9-1.20 | A-3 |

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in a polyethylene bag of not less than 50 micron thickness and then put either in a mild steel drum or a high density polyethylene container.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

- Name of the material;
- Name of the manufacturer and his recognized trade-mark, if any;
- Gross and net mass;
- Date of manufacture; and
- Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(Clause 2.2 and Table 1)

METHODS OF TEST FOR PHOSPHOROUS PENTACHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless otherwise specified, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect results of analysis.

A-2. DETERMINATION OF PHOSPHOROUS PENTACHLORIDE

A-2.1 Two methods, namely, acidimetric and argentometric methods have been prescribed for this determination. Argentometric method shall be the referee method.

A-2.1.1 *Reagents*

A-2.1.1.1 *Standard sodium hydroxide* — 1 N.

A-2.1.1.2 *Standard sulphuric acid* — 1 N.

A-2.1.1.3 *Methyl orange indicator*

A-2.1.1.4 *Standard silver nitrate* — 0.1 N.

A-2.1.1.5 *Standard ammonium thiocyanate* — 0.1 N.

A-2.1.1.6 *Ferric ammonium sulphate* — 40 percent.

A-2.1.1.7 *di-n Butyl phthalate*

A-2.2 **Procedure**

- a) *Acidimetric* — Accurately weigh about 1.2 g in a sealed ampoule. Place the ampoule in 50.0 ml of N sodium hydroxide contained in a stoppered 250-ml flask. Break the ampoule below the surface of the liquid and shake, set aside for 10 minutes, remove the stopper carefully, rinsing with a little water. Heat the flask on a steam bath for 30 minutes. Cool and titrate with N sulphuric acid using methyl orange indicator to a grey end point. Retain the solution for argentometric method, if required (b).

*Specification for water for general laboratory use (second revision).

- b) *Argentometric* — Dilute the contents of the flask retained from acidimetric method (a) to 500.0 ml with water. To 50.0 ml, add 10 ml of nitric acid, 50.0 ml of 0.1N silver nitrate, 3 ml of di-n-butyl phthalate and shake vigorously for 1 minute. Titrate the excess 0.1N silver nitrate with 0.1N ammonium thiocyanate using ammonium ferric sulphate as indicator shaking vigorously between each successive addition of titrant.

A-2.3 Calculation

- a) *Acidimetric*

$$\begin{array}{l} \text{Phosphorous pentachloride (as } \text{PCl}_5 \text{),} \\ \text{percent by mass} \end{array} = \frac{(V_1 - V_2) \times 3.47}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide added,

V_2 = volume in ml of standard hydrochloric acid, and

M = mass in g of the sample taken for test.

- b) *Argentometric*

$$\begin{array}{l} \text{Phosphorous pentachloride (as } \text{PCl}_5 \text{),} \\ \text{percent by mass} \end{array} = \frac{(V_1 - V_2) \times 4.16}{M}$$

where

V_1 = volume in ml of standard silver nitrate solution,

V_2 = volume in ml of standard ammonium thiocyanate, and

M = mass in g of sample.

A-3. BULK DENSITY

A-3.1 Procedure

A-3.1.1 Rest the funnel over the top edge of the tared graduated cylinder. Fill the cylinder to the 100-ml mark by pouring the material through the funnel, and drop it 20 times through a height of 15 cm on to a felt pad resting on a hard surface. Note the volume of the material after compacting and weigh the filled cylinder.

A-3.2 Calculation

$$\text{Bulk density, g/ml} = \frac{M}{V \times 1\,000}$$

where

M = mass in g of the material in the cylinder, and

V = volume in ml occupied by the material after compacting.

APPENDIX B

(Clause 4.1)

SAMPLING OF PHOSPHOROUS PENTACHLORIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS

B-1.1 For general requirements of sampling, the methods given in IS : 8883 (Part 1)-1978* may be followed.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment, all the drums, carbuoys or containers of the same size and of the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or different sizes of containers, the containers belonging to the same batch and same size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot separately. For this purpose, the number of containers to be selected from a lot shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

| LOT SIZE | NUMBER OF CONTAINERS/ CARBUOYS/DRUMS TO BE SELECTED |
|---------------|---|
| (1) | (2) |
| Up to 50 | 3 |
| 51 to 150 | 4 |
| 151 to 300 | 5 |
| 301 and above | 7 |

*Methods of sampling chemicals and chemical products : Part 1 General requirements and precautions.

IS : 11744 - 1986

B-2.3 The containers shall be selected from the lot at random and in order to ensure the randomness of selection, the method given in IS : 4905-1968* may be followed.

B-3. CRITERIA FOR CONFORMITY

B-3.1 The material shall be visually examined for the requirements given in 2.1 of this standard.

B-3.2 From each of the containers selected, adequate material shall be collected for determining the assay percentage.

B-3.3 On the basis of the test results of all the containers, the average (\bar{X}) and range (R) shall be calculated as follows:

$$\text{Average } (\bar{X}) = \frac{\text{Sum of the test results}}{\text{Number of tests}}$$

$$\text{Range } (R) = \text{Difference between the maximum and the minimum of the test results}$$

B-3.4 The lot shall be declared as conforming to the requirements of this standard, if

$$\bar{X} - 0.6 R \geq \text{the minimum requirements given in Table 1.}$$

*Methods for random sampling.

(Continued from page 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

| <i>Quantity</i> | <i>Unit</i> | <i>Symbol</i> |
|---------------------------|-------------|---------------|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

Supplementary Units

| <i>Quantity</i> | <i>Unit</i> | <i>Symbol</i> |
|-----------------|-------------|---------------|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

Derived Units

| <i>Quantity</i> | <i>Unit</i> | <i>Symbol</i> | <i>Definition</i> |
|----------------------|-------------|---------------|---------------------------------|
| Force | newton | N | 1 N = 1 kg.m/s ² |
| Energy | joule | J | 1 J = 1 N.m |
| Power | watt | W | 1 W = 1 J/s |
| Flux | weber | Wb | 1 Wb = 1 V.s |
| Flux density | tesla | T | 1 T = 1 Wb/m ² |
| Frequency | hertz | Hz | 1 Hz = 1 c/s (s ⁻¹) |
| Electric conductance | siemens | S | 1 S = 1 A/V |
| Electromotive force | volt | V | 1 V = 1 W/A |
| Pressure, stress | pascal | Pa | 1 Pa = 1 N/m ² |